SPECTROSCOPIC AND MAGNETIC STUDIES OF ERBIUM DOPED PHOSPHATE GLASS EMBEDDED WITH NATURAL FERRITE OXIDE NANOPARTICLES

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To my beloved father (H.Abu Bukhori) and my mother (Almh. Icah Napisah)
To my brothers and sisters (Denny, Robby, Fitri, Faiz and Tita)
for their enduring love, motivation and support

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ABSTRACT

Erbium doped zinc phosphate glass embedded with natural Fe_3O_4 nanoparticles having a composition of $(69\text{-}x)P_2O_5$ - 30ZnO - $1Er_2O_3$ – $(x)Fe_3O_4$, where x = 0, 0.5, 1.0, 1.5, 2.0 mol% has successfully been prepared by melt quenching technique. It was found that the color of the glass varies from pink (without natural Fe_3O_4 nanoparticles) to dark brown (2 mol% natural Fe_3O_4 nanoparticles). The change in color indicates that the natural Fe_3O_4 nanoparticles has already incorporated in the unit structure of the forming glasses. The amorphous structures of the glass sample were evident by the XRD analysis. The density and molar volume of the glass samples were found in the range of 2.75 − 2.89 g/cm^3 and 45.89 − 44.30 cm^3, respectively. The optical absorption edge was studied by using the UV-Vis spectroscopy. The optical band gaps for direct and indirect transitions and also Urbach energy varied in the range of 4.454 – 3.349 eV, 4.229 – 3.278 eV, and 0.637 – 0.233 eV, respectively were found to decrease with the increase of natural Fe_3O_4 nanoparticles contents. The emission spectrum was recorded using the photoluminescence spectrometer at room temperature. The emission spectra for 478 nm excitations displayed two prominent peaks centered at 532 nm and 634 nm that originate from $^4S_{3/2} \rightarrow {}^4I_{15/2}$ and $^4F_{9/2} \rightarrow {}^4I_{15/2}$ transitions, respectively. In addition, the intensity of luminescence spectra tends to quench the emission band with the increasing concentration of the natural Fe_3O_4 nanoparticles. The crystallite size of natural Fe_3O_4 nanoparticles was calculated using Debye-Scherrer formula and the results were compared with the size of nanoparticles obtained from TEM. The average diameter of the Fe_3O_4 nanoparticles of about 26 - 31 nm was calculated using Debye-Scherrer method and this value was about the same with that obtained by TEM micrographs. The TEM micrograph revealed the presence of spherical and homogenous distribution of natural Fe_3O_4 nanoparticles. Vibrating sample magnetometer (VSM) measurement showed that magnetization of glass increased with applied fields and showed paramagnetic and ferrimagnetic behavior.

ABSTRAK

Kaca zinc fosfat berdop erbium yang tersisip dengan nanopartikel Fe_3O_4 semulajadi dengan komposisi molar (69-*x*)P_2O_5 - 30ZnO - 1Er_2O_3 - (*x*)Fe_3O_4, dengan *x* = 0, 0.5, 1.0, 1.5, 2.0 mol% telah berjaya disediakan menggunakan teknik pelindapan leburan. Warna kaca didapati berubah daripada merah jambu (tanpa nanopartikel Fe_3O_4 semulajadi) ke coklat tua (2 mol% Fe_3O_4 semulajadi). Perubahan warna menunjukkan bahawa nanopartikel Fe_3O_4 semulajadi telah menyatu dalam struktur unit pembentukan kaca. Sifat amorfus sampel kaca telah disahkan oleh analisis XRD. Ketumpatan dan isipadu molar masing-masing adalah dalam julat 2.75 − 2.88 g/cm^3 dan 45.89 − 44.29 cm^3. Pinggir serapan optik dikaji menggunakan spektroskopi ultralembayung cahaya nampak. Jurang tenaga optik untuk peralihan terus dan tak terus serta tenaga Urbach masing-masing berada dalam julat antara 4.454 − 3.349 eV, 4.229 − 3.278 eV, dan 0.637 – 0.233 eV. Nilai ini didapati berkurang dengan pertambahan nanopartikel Fe_3O_4 semulajadi. Spektrum pancaran telah di rekod menggunakan spektrometer fotopendarcahaya pada suhu bilik. Spektrum pancaran diujakan pada 478 nm, menunjukkan kewujudan dua puncak yang berpusat di 532 nm dan 634 nm berasal dari transisi $^4S_{3/2} \rightarrow {}^4I_{15/2}$ and $^4F_{9/2} \rightarrow {}^4I_{15/2}$. Disamping itu, keamatan spektra pendarcahaya cenderung untuk mengurangkan jalur pancaran dengan pertambahan kandungan nanopartikel Fe_3O_4 semulajadi. Saiz hablur nanopartikel Fe_3O_4 semulajadi telah diperoleh menggunakan formula Debye-Scherrer dan dibandingkan dengan saiz nanopartikel yang diperoleh daripada mikroskop transmisi elektron (TEM). Nilai purata diameter nanopartikel Fe_3O_4 yang diperoleh ialah 26 – 31 nm dan nilai ini telah dikira menggunakan kaedah Debye-Scherrer didapati hampir sama dengan nilai yang dihasilkan oleh mikrograf TEM. Taburan mikrograf TEM menunjukkan kehadiran bentuk sfera dan taburan homogen nanopartikel Fe_3O_4 semulajadi. Pengukuran magnetometer sampel bergetar (VSM) menunjukkan bahawa pemagnetan kaca bertambah dengan medan yang dikenakan, serta menunjukkan perlakuan bahan paramagnetik dan ferimagnetik.

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LIST OF ABBREVIATIONS

Al_2O_3	-	Alumunium Oxide
BO	-	Bridging Oxygen
DC	-	Down-Conversion
Er^{3+}	-	Erbium
ET	-	Energy Transfer
fcc	-	Face Centered Cubic
FeO	-	Wustite
Fe_3O_4	-	Ferrite Oxide or Magnetite
α-Fe_2O_3	-	Hematite
γ-Fe_2O_3	-	Maghemite
FWHM	-	Full Length at Half Maximum
HR-	-	High-Resolution
HRTEM	-	High Resolution Transmission Electron Microscope
JCPDS	-	Joint Committee for Powder Diffraction Standards
JO	-	Judd-Ofelt
MgO	-	Magnesium Oxide
NBo		Non-Bridging Oxygen
NPs	-	Nanoparticle
NR	-	Non-Radiative
Oe	-	Oersted
P_2O_5	-	Phosphorus pentaoxide
P		Phosphorous atom
PL	-	Photo-Luminescence
RE	-	Rare Earth
TEM	-	Transmission Electron Microscope

UTM	-	Universiti Teknologi Malaysia
UV	-	Ultraviolet
VBM	-	Valence Band Maxima
VIS	-	Visible
VSM	-	Vibrating Sample Magnetometer
XRD	-	X-Ray Diffraction
ZnO	-	Zinc Oxide

LIST OF SYMBOLS

A	-	Absorption Coefficient
2θ	-	Angle of Diffraction
B	-	Magnetic Induction
d	-	Size of Nanoparticle, Thickness of the Sample
e	-	Charge of Electron
E	-	Electric Field
E_{dir}	-	Direct Optical Band Gap
E_{ind}	-	Indirect Optical Band Gap
ΔE, E_U	-	Urbach Energy
f	-	Oscillator Strength
H	-	Magnetic Field
I	-	Intensity
l	-	Length
J	-	Orbit Angular Momentum
m	-	Mass of Electron
n_2	-	Non-linear Refractive Index
M	-	Average Molecular Weight
N	-	Concentration
N_A	-	Avogadro's number
R	-	Glass Constant
S	-	Stability Factor
T'	-	Transmission
T	-	Temperature
T_c	-	Crystallization Temperature
T_g	-	Glass Transition Temperature

T_m	-	Melting Temperature
T	-	Time
V	-	Molar Volume
W	-	Weight
α_m	-	Polarizability
β	-	Branching Ratio
E	-	Dielectric Function
X	-	Susceptibility
P	-	Density
λ	-	Wavelength
T	-	Lifetime
S	-	Spin
L	-	Orbit
ρ	-	Density
w_a	-	Weight of the glass sample in air
w_b	-	Weight of the glass sample in liquid
ρ_b	-	Density of Tolluene
E_f	-	Energy of the final state
E_i	-	Energy of electron in lower band
B	-	Band tailing
$h\upsilon$	-	Photon energy
τ_R	-	Radiative lifetime
η_R	-	Luminecent efficiency
τ_{NR}	-	Non-radiative lifetime
M	-	Magnetic moment
H_c	-	Coercivity
Mr	-	Remanent magnetization
Ms	-	Saturation magnetization
Mr/Ms	-	Squarness

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Glasses containing rare earth ions and embedded with magnetic nanoparticles (NPs) are getting large attention because the presence of NPs which may lead to the increase of luminescence intensity, thermal stability (Widanarto *et.al.,* 2013) and magnetic responsibility (Piaoping *et.al.,* 2009) (Hongxia *et.al.,* 2012). Among the current interest material, the P_2O_5 based glasses have attracted a large attention due to their various applications in optical data transmission, sensing and laser technologies. The characteristics of phosphate glasses include high transparency, low melting point, high thermal stability, and high gain density which is attributed to the high solubility of rare earth ions, low refractive index and low dispersion (Yung *et.al.,* 2011) (Das *et.al.,* 2009).

The choice of host glasses materials are very important in the development of optical devices doped with rare earth (RE). Among different glass host matrices, phosphate glasses have unique advantages over the other glass host matrices. Phosphate glasses matrices with suitable compositions have been found to be good hosts for different laser active ions (Talib *et.al.*, 2008). The properties of phosphate

glasses such as relatively high thermal expansion coefficients, low optical dispersions and low glass transition temperatures compare with their silicate or borate counterparts, make them technologically important material in spite of their hygroscopic and volatile properties. The hygroscopic behaviour of phosphate glasses and the volatility of P_2O_5 at elevated temperature are two of the main reason for the limited number of investigations on the structure and the properties of these glasses. However, due to all the good properties of phosphate glass, they are hygroscopic and the OH^- group which are present and acting like high energy phonons can severely affect the quantum efficiency of the emitting RE^{3+}. The main source of OH^- group is due to the starting material and the atmospheric moisture during the melting process hence there are some ways to reduce its incorporation in the glass by variations of composition and procedure (Andrea *et.al.,* 2008).

Glasses of the Er^{3+} have been reported to exhibit the largest magnetic contribution to the low temperature specific heats known in oxide glasses. Natural iron oxides, which are mostly in the form of magnetit (Fe_3O_4), hematite (α-Fe_2O_3) and maghemite (γ-Fe_2O_3) can be easily found in iron sand. The α-Fe_2O_3 has the same spinel structure as magnetite but has no divalent ions. The magnetic Fe^{3+} ions are positioned in two sublattices with different oxygen coordination. The ferrimagnetism arises from the unequal distribution of these ions in A and B sites. Recently, a low temperature spin glass transition was found at T = 42 K. The γ-Fe_2O_3 has a corundum crystal structure and has an antiferromagnetic order below 950 K, while above the Morin point (260 K) it exhibits so called weak ferromagnetism. Below melting temperature (T_m) the two magnetic sublattices are oriented along the rhombohedral [111] axis. The spin canting results in a weak ferromagnetic in the (111) plane. Hematite is the most stable iron oxide (Elena., 2004).

Incorporation of iron oxides into the glass has paramount importance due to their excellent magnetic, optical and electrical properties (Van., 1999). Magnetite (Fe_3O_4) is an important magnetic material that is widely used for different

applications such as magnetic sensors, printing ink and catalysts which especially in the biomedical field (Mazlini *et.al.,* 2012).

1.2 Problem Statement

The used of iron oxides (FeO, Fe_2O_3 and Fe_3O_4) as dopant has provide interesting phenomena over the structure and the properties of the phosphate glasses. In addition the iron-doped glasses exhibit interesting electrical and magnetic properties that depend on the iron oxide species and their coordination symmetry (Hussin, 2011). Fe_3O_4 nanoparticles are common ferrite oxides with an inverse spinal structure (Vijaya *et.al.,* 2005). These class of compounds exihibit unique electrical and magnetic properties due to the electron transfer between Fe^{2+} and Fe^{3+} in tetrahedral and octahedral sites. In addition, the transition metal ions will contribute to a multivalence state in the glass which influence the properties (Ensanya *et.al.,* 2009).

The effect of natural Fe_3O_4 nanoparticles (NPs) especially in the enhancement of emission intensity has been studied by few workers. Table 1.1 summerized the list of previous researchers regarding the studies on magnetic properties of glasses. However, there is a very limited study on the optical and magnetic properties of glass containing natural Fe_3O_4 NPs. Thus, it is the aim of this study to focus on both optical and magnetic properties in order to find their properties. It is expected that this study can provide more informations on the basic knowledge of the glass system.

Table 1.1 : The list of previous reseachers

Researcher	Title	Glass preparation technique	Material composition
Maximina Romero, *et. al.,* (2001)	Magnetic properties of glasses with high iron oxide content	Melt quenching technique	Fe_2O_3, SiO_2, Al_2O_3, CaO, MgO, ZnO, PbO, Na_2O, K_2O
I.S. Edelman, *et. al.,* (2001)	Superparamgnetic and ferrimagnetic nanoparticles in glass matrix	Melt quenching technique	Fe_2O_3, MnO, Li_2O, BaO
Mekki, *et.al.,* (2006)	Structural and magnetic investigation of Fe_2O_3-TeO_2 glasses	Melt quenching technique	Fe_2O_3 and TeO_2
N.A. Zarifah, *et.al.,* (2010)	Magnetic behaviour of $(Fe_2O_3)_x$ $(TeO_2)_{1-x}$ glass system due to iron oxide	Melt quenching technique	Fe_2O_3 and TeO_2
J. Kaewkhao, *et.al.,* (2010)	Structural and magnetic properties of glass doped with iron oxide	Melt quenching technique	Na_2O, Al_2O_3, B_2O_3, CaO, Sb_2O_3, BaO, Fe_2O_3
Ping Hu, *et.al.,* (2011)	Heat treatment on Fe_3O_4 nanoparticles structure and magnetic properties prepared by carbothermal reduction	Carbothermal reduction method	Fe_3O_4, NH_3, H_2O
H. El. Ghandor	Synthesis and some physical propertis of magnetite (Fe_3O_4) nanoparticles	Co-precipitation method	Fe_3O_4, NH_4, SO_4 and $FeCl_3$.

1.3 Objectives of the Research

The objectives of the study are:

i) To optimise the preparation of the Er^{3+} doped phosphate glasses of the $(69\text{-}x)P_2O_5\text{-}30ZnO\text{-}1Er_2O_3\text{-}(x)Fe_3O_4$ system (x = 0, 0.5, 1.0, 1.5 and 2.0 mol %).

ii) To characterize the amorphous nature by using x-ray diffraction (XRD)

iii) To determine the physical properties of the glass.

iv) To characterize the glass by transmission electron microscope (TEM) imaging, UV-VIS and photoluminescence (PL) spectroscopy.

v) To characterize the magnetic properties and the effect of nanoparticles.

1.4 Scope of Study

In order to achieve the above objectives the works have been focused on the given scope :

i. Ferrite oxide (Fe_3O_4) obtained from natural iron sand.
ii. Preparation of glass based on the $(69\text{-}x)P_2O_5\text{-}30ZnO\text{-}1Er_2O_3\text{–}(x)Fe_3O_4$ system (x = 0, 0.5, 1, 1.5 and 2 mol %) using the melt quenching technique.
iii. Characterization of amorphous nature by using XRD.
iv. Determination of the density and molar volume of the glass system.
v. Characterization of absorption by using UV-VIS-NIR spectrophotometer.
vi. Characterization of emission by using Photoluminescence spectrophotometer.
vii. Characterization of morphology of nanoparticles by using Transmission Electron Microscopy.
viii. Characterization of magnetic properties by using Vibrating Sample Magnetometer.

1.5 Choice of System

To achieve the objectives of the study, a series of the glass system were chosen as below:

$(69\text{-}x)P_2O_5\text{-}30ZnO\text{-}1Er_2O\text{-}(x)Fe_3O_4$, where x is $0.0 \text{ mol \%} \leq x \leq 2.0 \text{ mol \%}$

Phosphate glasses shows several advantages over conventional silicate and borate glasses due to their superior physical properties such as low melting and softening temperatures and high ultra-violet (UV) and far infrared transmission (Ensanya *et.al.,* 2009) (Khor *et.al.,* 2012). Important characteristics such as relatively high thermal expansion coefficients, low optical dispersions and low glass transition temperatures make them highly suitable for many applications. However, their very high hygroscopic nature due to excessive amount of OH^- contents limit the quantum efficiency of the emitting RE^{3+} ions which is decisive for device performance. The OH^- groups that mainly originates from the starting material and the atmospheric moisture during the melting process can considerably be reduced by incorporating suitable modifier such as ZnO and MgO in the host (Andrea *et.al.,* 2008) (Wiench *et.al.,* 2000).

There has been a continued interest among researcher in zinc phosphate glass system for various of application. The presence of zinc, which acting as a network former/modifier imparts better chemical durability, low glass transition temperatures and wider glass forming composition range. Due to higher transparency of zinc phosphate glasses in the ultraviolet region compared to conventional silicate glasses, UV light absorbing materials can be incorporated in these glasses (Dheeraj *et.al.,* 2009).

Iron oxide (Fe_3O_4) used as dopant provide interesting phenomena over structure and the properties of the phosphate glasses (Elisa *et.al.,* 2005). On the other hand, the iron-doped glasses exhibit interesting electrical and magnetic properties

due to the electron transfer between Fe^{2+} and Fe^{3+} in tetrahedral and octahedral sites. Here the electron Fe^{3+} moves to interface where it undergoes a one electron reduction to form electron Fe^{2+}. Futhermore, the transition metal ions will contribute multivalence state in the glass and dramatically influence the properties (Widanarto *et.al.,* 2013).

In recent times, glasses containing rare earth ions and embedded with magnetic nanoparticles are getting large attention because the presence of the NPs, which may lead to the increase of luminescence intensity, thermal stability and magnetic responsibility (Widanarto *et.al.,* 2013) (Piaoping *et.al.,* 2009) (Hongxia *et.al.,* 2012).

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